UCN-2383 (3 11-60)

ORNL OAK RIDGE NATIONAL LABORATORY STER COPY

UNION CARBIDE NUCLEAR COMPANY Division of Union Carbide Corporation



Post Office Box X Oak Ridge, Tennessee INTERNAL USE ONLY

ORNL Het

CENTRAL FILES NUMBER

62-1-38

COPY NO. 26

DATE:

January 29, 1962

SUBJECT: Low Level Waste Treatment Pilot Plant -

Run HR-3 Summary

TO:

F. L. Culler, Jr.

FROM:

R. E. Brooksbank

ABSTRACT

The results are reported of the second demonstration run, HR-3. using the scavenging-ion exchange process for decontaminating low level activity water. The run time was extended by an additional 500 resin bed volumes, over previous runs, increasing the total to 2,086 bed volumes. A total of 96 hours of continuous operation was accumulated to process 58,400 gallons of ORNL waste solutions. During the run, the Sr90 decontamination factors ranged from 6100 (1500 bed volumes) to 2047 (2086 bed volumes). Cs137 decontamination factors ranged from 788 (1500 BV) to 246 (2086 BV) during the same period. Plant effluent contained less than 1.5 per cent of current MPCw values for these isotopes for a 168-hour week.

> This document has been approved for release to the public by:

NOTICE

This document contains information of a preliminary nature and was prepared primarily for internal use at the Oak Ridge National Laboratory. It is subject to revision or correction and therefore does not represent a final report. The information is not to be abstracted, reprinted or otherwise given public dissemination without the approval of the ORNL patent branch, Legal and Information Control Department.

1.0 INTRODUCTION

A scavenging-ion exchange process (1) which will decontaminate large volumes of slightly contaminated water has been demonstrated in laboratory scale equipment. (2) Following these studies, a 10 gpm pilot plant was designed (3) and constructed in order to demonstrate the economic, chemical and equipment phases of the process. This report is the second in a series of run summaries describing the results obtained in the Low Level Waste Treatment Pilot Plant during the treatment of ORNL process waste water. It will serve to transmit recent information to those interested as well as to document the results obtained.

The flowsheet followed includes adjusting the pH of the waste water to 11.8 with NaOH followed by the addition of ferrous sulphate to form a light floc. The resulting precipitate, consisting of insoluble metallic carbonates as well as floc, is removed from the system by percolation through a dense sludge blanket in the bottom of the clarifier vessel. Bulk quantities of dissolved and suspended materials along with a significant fraction of the radioactive contaminants are removed in the precipitate.

Effluent from the clarifier vessel is passed through a polishing filter for the further removal of hardness and turbidity and then through a bed of Duolite CS-100 cation ion exchange resin which removes the remaining hazardous contaminants, largely strontium and cesium. The material sorbed on the resin bed (a phenolic-carboxylic type) is periodically eluted with 0.5 $\underline{\text{M}}$ HNO3; after washing the bed with water it is converted to the sodium form with 0.1 M NaOH.

Specific objectives of the second run, HR-2, were to:

- l. Compare the fission product decontamination factors obtained with those in the laboratory, the semi-pilot plant and previous pilot plant runs. $^{(4)}$
- 2. Compare the use of 5 ppm iron coagulant with 10 ppm as used during run HR-2. $^{(4)}$
 - 3. Study the effect of increased resin column on-stream time.

- 4. Establish the feasibility of the split-resin column elution procedure which will reduce the process waste volume.
 - 5. Observe resin column loading characteristics.
- 6. In general, observe the operating characteristics of the pilot plant components.

2.0 SUMMARY

A total of 58,420 gallons of ORNL process waste water was processed through the Low Level Waste Treatment Pilot Plant using the scavenging-ion exchange process during a 96-hour continuous operating period.

After 1500 resin column bed volumes had been treated, the over-all Sr⁹⁰ and Cs¹³⁷ decontamination factors were 6143 and 788, respectively. Based on a radiation scan of the resin bed and the absence of a calcium breakthrough, it was decided to extend the on-stream life of the resin. After 2086 total bed volumes the Sr⁹⁰ and Cs¹³⁷ decontamination factors were 2047 and 246 respectively, representing greater than 99.5 per cent removal for both isotopes. Analysis of plant effluent indicated a product which was 1.4 and 0.04 per cent, respectively of current MPC_w values for Sr⁹⁰ and Cs¹³⁷ for a 168-hour week.

Of the total $\rm Sr^{90}$ removed, 53 per cent was removed during the precipitation and clarification steps. As in the previous run, the remainder of the $\rm Sr^{90}$ was removed by ion exchange. Essentially no $\rm Cs^{137}$ was removed by precipitation; the total was removed by ion exchange.

The use of a "split elution" cycle was attempted after HR-3. The tail-end (5 resin bed volumes) solution resulting from a previous elution was used as the original elutriant for fission product desorption. The objective of this treatment was to reduce the volume of waste resulting from the process. No adverse effects were noted as > 99.9% of the Sr and Cs was removed from the resin after 10 bed volumes.

The rotary drum sludge filter did not perform adequately, making the use of the decantation vessels necessary. Continuous adjustment of the variables affecting the unit was necessary requiring excessive operator effort. A pressure filter has been recommended as a replacement for the rotary unit.

The calculation and tabulation of process data was speeded with the aid of a Fortran program for use in the IBM 7090 computer.

3.0 TABULATION OF RESULTS

3.1 Feed

A total of 58,420 gallons of process waste water representing 2086 ion exchange bed volumes, was pumped into the facility. The feed was withdrawn from the suction leg of the Lime Soda plant feed pump which is supplied by the 1,000,000 gallon equilization basin.

The average hardness (as CaCO₃) of the untreated feed was 124 ppm; pH values ranged from 6.4 to 7.3 and averaged 6.9.

Radiochemical analysis of composited feed solutions are presented in Table 3.1.

Gross β	(c/m/ml)	17.3
Gross γ	(c/m/ml)	15.6
Sr ⁹⁰ β	(d/m/ml)	61.43
Sr ⁸⁹	(d/m/ml)	$_{\mathrm{N.D.}}^{\mathrm{(a)}}$
co ⁶⁰	(d/m/ml)	14.32
Ru ¹⁰⁶	(d/m/ml)	N.D. (a)
TRE	(d/m/ml)	5.10
Cs ¹³⁷	(d/m/ml)	39.42
Zr-Nb	(d/m/ml)	3.41
I-131	(d/m/ml)	N.D.

Table 3.1 Run HR-3 Radiochemical Analysis of Feed

3.2 Decontamination and Fission Product Removal

The major contaminant and health hazard, Sr^{90} , was removed from the feed by a factor which ranged from 2047 (2086 bed volumes) to 6143 (1500 bed volumes). Cesium removal during the same operating period ranged from 246 to 788. Plant effluent contained less than 1.5 per cent of current MPC values for Sr^{90} and Cs^{137} based on a 168-hour week.

Of the total Sr⁹⁰ removed, 53 per cent was collected in sludge blanket in the bottom of the clarifier and the remainder was collected

⁽a) Analyzed for but not detected.

on the cation exchange column. As in the previous run, essentially all of the (99.8%) ${\rm Cs}^{137}$ was removed by ion exchange.

A breakdown of the removal of the activities and isotopes during major process steps is presented in Table 3.2.

Table 3.2 Activity Removal - Run HR-2

	Across Precipita	ation	Across	Ion Exchange		
	Clarification-f	iltration		% Removed	Over-all	Process
Activity	DF 9	Removed	DF	(of remaining)	DF	% Removed
Gross β	3.09	67.63	14.74	93.21	45.53	97.80
Gross y	1.67	40.09	15.29	93.46	25.51	96.08
$\text{Sr}^{90} \beta^{(1)}$	2.13	52.95	963.33	99.90	2047.67	99•95
Sr^{90} $\beta^{(2)}$	2.13	52.95	2890.0	99.97	6143.00	99.98
Sr ⁸⁹	N.D. (3)					
co ⁶⁰	14.61	93.16	ı	0	11.46	91.27
Ru ¹⁰⁶	N.D. (3)					
TRE	4.18	76.08	6.78	85.25	28.33	96.47
Cs ¹³⁷ (1)	1	0	276.75	99.64	246.37	99.59
$Cs^{137}(2)$	l	0	885.60	99.89	788.40	99.87
Zr-Nb	3.63	72.43	N.C.	> 99•9	N.D.	> 99.9
r ¹³¹	N.D. (3)	des emp				~ ~

⁽¹⁾ After 2086 resin bed volumes

4.0 FLOWSHEET

4.1 Chemical Flowsheet

The standard flowsheet used to effect the decontamination is shown in Fig. 4.1. During Run HR-3 5 ppm Fe as FeSO₄ · 7H₂O was added rather than the 10 ppm requirement used during Run HR-2. The only noticeable chemical effect of this change was an increase in the TRE decontamination (71.6% removed during HR-2 as opposed to 96.5% during HR-3).

The original flowsheet as planned was satisfactorily operated for 1500 bed volumes; however because of the results of γ radiation scans (Section 5.4.1) and the hardness titrations of the resin effluent stream, it was decided to increase the on-stream life of the column.

⁽²⁾ After 1500 resin bed volumes

⁽³⁾ None detected

~ 30 min holdup FLOCCULATOR

FLASH MIXER

waste 5 ppm in iron Copperas to make

~ 3 vol sludge

FILTER

2-3 hr holdup

CLARIFIER

PROCESS WATER WASTE

per 3.5-7 day cycle

NaOH to make waste 0.01 M

Fig. 4.1. Process water decontamination with carboxylic-phenolic ion-exchange resin.

~ 0.1 vol dry cake

STORAGE

4.2 Equipment Flowsheet

The equipment flowsheet used in the plant is shown in Fig. 4.2.

5.0 PROCESS PERFORMANCE

5.1 Flash Mixer and Flocculation

No adverse effects were noted in the flocculation step, using 5 ppm iron rather than 10 ppm. The indicated pH of the solution in the flocculator vessel averaged 11.86; analysis of the solution by a Beckman pH meter gave values which averaged 11.96. The flow of concentrated NaOH to the flash mixer was constant within ±3%.

5.2 Clarifier

A total of 96 hours of continuous operation was completed using the 1870-gallon clarifier vessel. The coagulant rate was doubled during startup to permit the rapid formation of a sludge blanket. The hardness of the clarifier effluent ranged from 35 to 70 ppm (as CaCO₃) and averaged 57 ppm. Turbidity of this stream averaged 5 ppm.

5.3 Polishing Filters

Both sand and anthracite polishing filters were required during HR-3. The sand filter was removed from service after 71 hours of operation because of excessive effluent hardness (maximum of 16 ppm) and pressure drop (maximum of 12.8 psi). The anthracite filter had increased only 0.5 psi as a result of accumulated solids during the remainder of the run. The average turbidity of the effluent from sand filtration was 0.6 ppm; 1.25 ppm was obtained during coal filtration.

Back wash water for filter regeneration was recycled to the system.

5.4 Ion Exchange

A total of 2086 bed volumes of treated waste water was passed through the 10-in. diameter by 8-ft resin bed. Effluent from the unit contained no detectable hardness; the turbidity was 2.13 which was due to lightly colored organic substances leached from the resin. The average pressure drop through the bed was 16.1 psig.

5.4.1 Resin Radiation Profile

The use of a movable γ radiation probe through the length of the resin bed proved to be an effective tool for anticipating resin breakthrough. Throughout Run HR-3, the location of the maximum radiation

UNCLASSIFIED ORNU-LE-DWG 59977 R1

band was noted as a function of resin bed volumes passed through the column. Of interest are the curves presented in Fig. 5.4.1 showing the radiation γ scans taken at 1500, 1757, 1928, and 2035 bed volumes. The double peak effect within the bed at 1757 volumes is probably due to the increased delivery of Ca to the resin columns. Laboratory studies have indicated that Ca is sorbed on CS-100 resin more strongly than either Sr 90 or Cs 137 . Shortly after 1500 bed values, the sand filter effluent increased in hardness from 8 to 16 ppm. The formation of the second radiation peak was established after the change was made to anthracite filtration.

5.4.2 Resin Column Elution

A total of 10 bed volumes of 0.5 $\underline{\text{M}}$ HNO $_3$ elutriant solution was passed up-flow through the bed for fission product desorption. The "split" elution cycle was used to reduce the volume leaving the process. In the "split" solution procedure, five volumes of elutriant solution from the tail-end of the previous elution is used as the initial elutriant. The second five volumes, containing essentially no fission product contamination, is then held in storage for subsequent elution cycles (see Fig. 5.4.2).

Decontamination factors* for Sr⁹⁰ and Cs¹³⁷ were greater than 10⁴ after 10 bed volumes. The removal of the twin activity bands from the column during HR-3 may be followed by the elution curves presented in Figures 5.4.2, 5.4.3, and 5.4.4.

6.0 SLUDGE FILTRATION

A total of 1,019 pounds of wet sludge was withdrawn from the system with the rotary drum filter and decantation vessels. This sludge was estimated to contain 50 per cent water by volume.

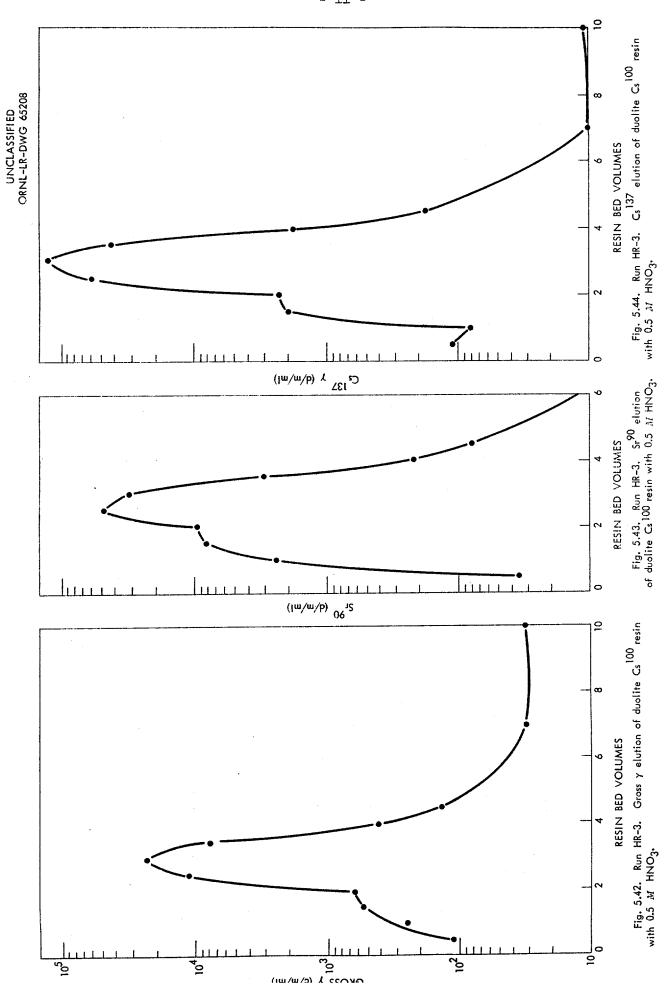
The capacity of the rotary drum filter was not adequate for use during HR-3, making the use of the decantation vessels mandatory. Considerable operator effort was expended in correcting frequent malfunctions of the unit. Variables that required adjustment through-

 $^{^*}$ DF = $\frac{\text{Peak concentration in eluent}}{\text{Concentration after 10 bed volumes}}$

.

DOWNFLOW DIRECTION - 10-in. dia

Fig. 5.41. Run HR-3. Resin radiation profile.



out the run included:

- (a) vacuum adjustment
- (b) filtrate removal rate
- (c) drum speed
- (d) feed pump rate
- (e) recycle rate
- (f) precoat thickness
- (g) distance of doctor blade from drum.

The operation of the Low Level Pilot Plant should require little manpower effort because of the simplicity of equipment. A suitable pressure filter with adequate containment for sludge removal has been recommended for replacement of the rotary filter.

7.0 DATA TABULATION

7.1 Fortran Program

A simple Fortran program has been written to speed the tabulation and calculation of process data. The data contained in this report and all subsequent reports will be handled by this method.

8.0 REFERENCES

- 1. J. T. Roberts and R. R. Holcomb, "A Phenolic Resin Ion Exchange Process for Decontaminating Low-Radioactivity-Level Process Water Wastes," ORNL-3036, May 22, 1961.
 - R. R. Holcomb and J. T. Roberts, "Low Level Waste Treatment by Ion-Exchange, II. Use of a Weak Acid, Carboxylic-Phenolic Ion-Exchange Resin," ORNL-TM-5, September 25, 1961.
- 2. R. E. Blanco and E. G. Struxness, "Waste Treatment and Disposal Progress Report for April and May, 1961," CF-61-7-3, August 1, 1961.
- 3. J. M. Holmes and W. R. Whitson, "Design of a Low Level Pilot Plant for Scavenging-Ion Exchange Process," (in preparation).
- 4. R. E. Brooksbank, "Low Level Waste Treatment Pilot Plant Run HR-2 Summary," December 21, 1961, CF-61-12-32.

Table 7.1 Low Level Waste Treatment - HR-3

1	1		ar.	l																										
		Ton	Exchange	12.00	•	11.80	1.88	•	11.85	11.92	•	11.91	11.85	11.85	•	11.80	11.90	11.90	11.80	11.82	•	•	•	11.92	•	•	•	11.82	11.80	11.80
Hu	1111		Clarifier	11.90	11.85	11.90	11.80	11.90	11.99	11.88	•	•	•	11.95		•	•	•		11.95			•	11.95			•		11.90	11.90
		Floccu-	lator	11.85	11.85	11.80	11.89	11.80	11.85	11.8	11.92	11.85	11.85	11.85	11.85	11.80	11.90	11.90	11.90	11.82	11.93	11.80	11.80	11.93	11.80	11.85	11.90	11.80	11.85	11.90
			Feed	7.06	6.95	7.13	7.25	7.10	7.20	2.00	6.80	6.85	2.00	7.20	7.10	7.10	7.10	7.10	7.00	6.80	7.10	7.10	8.8	ر د ا	9.60	€.15	o. 10,	9.60	6.70	o. 70
m)	Ton	Exchange	Column	4.0	1.0	٥٠٦	1.0	1.5	0.0	1.5	0°0	1.0	0.5	1.0	0.0	3.0	3.0	8.0	0°0	3.0	o• 8•0	0.0	0.4	٥ . ٣	1,5	3.5	3.0	o. 0	8.0	5.0
Turbidity (ppm		Polishing	Filter	0	0	0	0	0	0	0	0	0	0	0	0	3.0	1.0	1.0	٥.	1.0	1.0	0.1	0.8	1.0	0	•	7.0		•	0
Tur			Clarifier	•	3•0	0.4	5.0	•	•		0.9	•	•	0 . 8	•	•	0.9	•		•	0.9	•		0.9	4.0	5.0	5.5	5.0	5.0	0.0
)3)	10 TO		Colum	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	ભા	0	0	0	0	0	0 (ɔ
(ppm as CaCO ₃)		Polishing	Filter	ന		7	<u></u>	ω,	9	9	ω (∞	9	9'	9	<u></u>	ထ	φ (∞	ឧ	သ	점,) 16	16	*	7	on ;	10	;	†
Hardness (pp	1		Clarifier	26	35	23	62	09	, 52	65	1 9	o <u>;</u>	1 9	<u>0</u>	28	55	58	57	₹,	9'	δ.	23	57	נל	, 58 5	₽ ,	0 /	δ,	, 28 1	59
Ha			Feed	211	112	971	714	9116	114	118	118	120	114	116 10	718 9	911	114	118	021 150	128	126	12g	124	124	130	94	34.	β: :Ε	144	T-40
		Time,	hr.	•	•	•	12.5	•	•	•	•	•	•	•		•	•	•			•	•	•			•		•	8,	•

*Switched to W-24 Anthracite Filter

UCN-2383 (3 11-60)





OAK RIDGE NATIONAL LABORATORY

Operated by

UNION CARBIDE NUCLEAR COMPANY Division of Union Carbide Corporation



Post Office Box X Oak Ridge, Tennessee INTERNAL USE ONLY

CENTRAL FILES NUMBER

CF-61-12-32

DATE:

December 21, 1961

COPY NO. 23

SUBJECT: Low Level Waste Treatment Pilot Plant - Run HR-2 Summary

TO:

F. L. Culler, Jr.

FROM:

R. E. Brooksbank

ABSTRACT

The results of the first demonstration run using the scavangingion exchange process for decontaminating low level activity water are reported. A total of 73 hours of continuous operation was accumulated to process approximately 44,000 gallons of ORNL waste. Major contaminants, Sr^{90} and Cs^{137} were removed from feed solutions by factors of approximately 3000 and 300, yielding a plant effluent which was less than 1 per cent of current MPC values for these isotopes for a 168hour week.

NOTICE

This document contains information of a preliminary nature and was prepared primarily for internal use at the Oak Ridge National Laboratory. It is subject to revision or correction and therefore does not represent a final report. The information is not to be abstracted, reprinted or otherwise given public dissemination without the approval of the ORNL patent branch. Legal and Information Control Department.

This document has been approved for release to the public by:

R Hamin

1.0 INTRODUCTION

A scavenging-ion exchange process (1) which will decontaminate large volumes of slightly contaminated water has been demonstrated in laboratory scale equipment. (2) Following these studies, a 10 gpm pilot plant was designed (3) and constructed in order to demonstrate the economic, chemical and equipment phases of the process. This report, the first in a series of run summaries describing the results obtained in the Low Level Waste Treatment Pilot Plant during the treatment of ORNL process waste water, will serve to transmit recent information to those interested as well as to document the results obtained.

The flowsheet followed includes adjusting the pH of the waste water to 11.8 with NaOH followed by the addition of ferrous sulphate to form a light floc. The resulting precipitate consisting of insoluble metallic carbonates as well as floc is removed from the system by percolation through a dense sludge blanket in the bottom of a clarifier vessel. Bulk quantities of dissolved and suspended materials along with a significant fraction of the radioactive contaminants are removed with the precipitate.

Effluent from the clarifier vessel is passed through a polishing filter for the further removal of hardness and turbidity and then through a bed of CS=100 cation ion exchange resin which removes the remaining hazardous contaminants, largely strontium and cesium. The material sorbed on the resin bed (a phenolic-carboxylic type) is periodically eluted with 0.5 $\underline{\text{M}}$ HNO3; after washing the bed with water it is converted to the sodium form with 0.1 $\underline{\text{M}}$ NaOH.

Specific objectives of the first run, HR-2, were to:

- 1. Compare the fission product decontamination factors obtained with those obtained in the laboratory and in a semi-pilot plant.
- 2. Study the effect of 10 ppm iron rather than the 5 ppm iron flocculation agent used in semi-pilot plant tests.
- 3. Establish the nature of fission product removal from the resin bed in order to determine the fraction of spent elutriant that could be recycled to the process.
- 4. In general, observe the operating characteristics of the pilot plant components.

2.0 SUMMARY

A total of 44,425 gallons of ORNL process waste water was processed through the Low Level Waste Treatment Pilot Plant using the scavenging-ion exchange process.

The major contaminants, strontium and cesium were removed from the feed by factors of 2956 and 287 respectively during the 73 hours of continuous operation, during which time 1564 bed volumes were passed through the ion exchange column. Analysis of plant effluent samples indicated a product which was 0.9 and 0.009 per cent, respectively of current MPC values for Sr⁹⁰ and Cs¹³⁷ for a 168-hour week. Of the total strontium removed, 63 per cent was collected in the dense sludge blanket formed in the clarifier and the remainder was collected on the cation exchange resin. Essentially no cesium was removed during the precipitation step; the total removal was by ion exchange.

More than 99% of the Sr and Cs sorbed on from the 10-inch diameter CS-100 resin bed was removed in passing 4-5 bed volumes of 0.5 \underline{M} nitric acid through the bed and > 99.9% Sr and Cs was removed after 10 bed volumes.

In general, equipment operability throughout run HR-2 was good. Two operational difficulties were encountered; (1) an abundance of algae, dirt and crud during the early phases of the run and; (2) the inability of the rotary drum filter to keep up with the formation of sludge.

3.0 TABULATION OF RESULTS

3.1 Feed

A total of 44,425 gallons of process waste water representing 1564 ion exchange bed volumes, was pumped into the facility. The feed was withdrawn from the suction leg of the Lime Soda plant feed pump which is supplied by the 1,000,000 gallon equilization basin. Solution was pumped into the system for a period of 97 hours of which the first 24 hours was used to bring the clarifier sludge blanket to equilibrium prior to placing the resin column on stream.

The average hardness (as CaCO₃) of the untreated feed was 120 ppm; pH values ranged from 6.9 to 7.9 and averaged 7.5.

Radiochemical analysis of composited feed solutions are presented in Table 3.1.

Table 3.1. Run HR-2 Radiochemical Analysis of Feed (c/m/ml)16.5 Grγ (c/m/ml)Grβ 10.9 sr⁹⁰, sr⁸⁹ (d/m/ml)59.2 cs¹³⁷ (d/m/ml)11.5 (d/m/ml)TRE 5.6 _T131 (d/m/ml)< 1.0 (d/m/ml)Zr-Nb < 0.2 co⁶⁰

10.6

3.2 Decontamination and Fission Product Removal

The major contaminant, Sr⁹⁰, was removed from the feed by a factor of 2956 representing 99.9 per cent removal. Plant effluent contained 0.9 per cent of current MPC_w (4) values for $Sr^{89} + Sr^{90}$ based on a 168-hour week. A breakdown of the removal of the activities and isotopes during the major process steps is presented in Table 3.2.

(d/m/ml)

Table 3.2 Activity Removal-Run HR-2

enastrony money and a file about		Precipitation	Acros			. 7.7 D
Activity	DF DF	tion-filtration % Removed	DF	<pre>% Removed (of remaining)</pre>	Over- DF	all Process % Removed
Grβ	4.8	79.2	6.4	84.0	30.0	96.7
Gry	4.4	77.1	10.1	90.1	44.2	97.7
sr ⁹⁰ , sr ⁸⁹	2.7	63.1	1090	99•9	2956	99.9
Cs ¹ 37	1	0	288	99•5	287	99.5
_{Co} 60	19.1	92.1	1.3	22.6	16.3	93.9
TRE	3.5	71.1	1.0	1.8	3•5	71.6

4.0 FLOWSHEET

4.1 Chemical Flowsheet

The standard chemical flowsheet used to effect the decontamination is shown in Fig. 4.1. During Run HR-2 it was decided to add 10 ppm Fe as $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ to the flash mixer rather than 5 ppm as specified by the flowsheet. This change was deemed necessary at the time because of the abundance of nonferrous floc, dirt and algae in the flocculator which tended to rise to the surface. This condition was probably brought about by the processing of

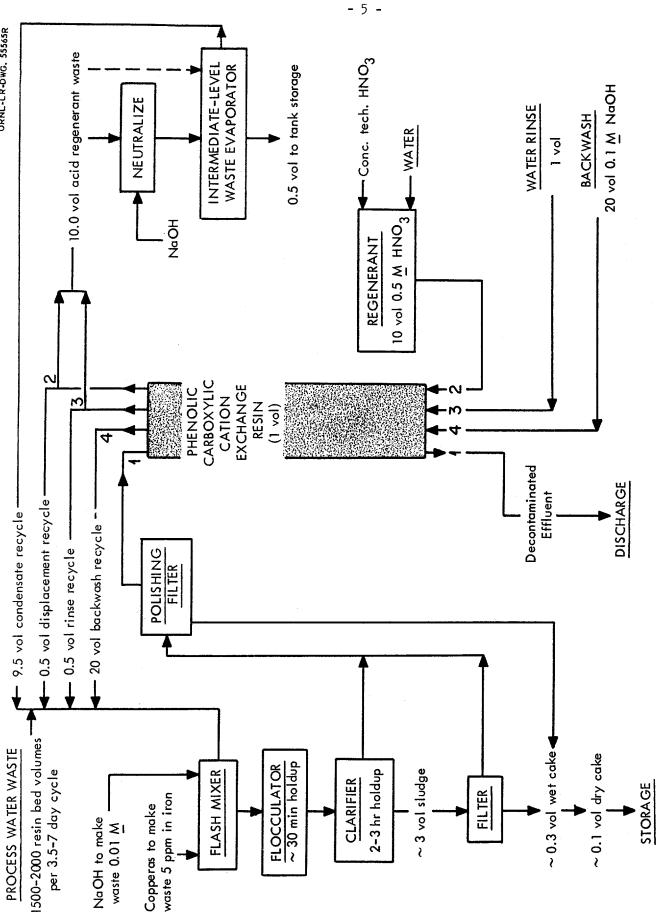


Fig. 4.1. Process water decontamination with carboxylic-phenolic ion-exchange resin.

solution that had remained stagnant in a "dead leg" in the feed line from the Lime Soda Plant. The line was apparently flushed clear of this material after 8 hours of operation, but no change was made in the $\text{FeSO}_{\text{l}_{\text{l}}}$ addition rate.

4.2 Equipment Flowsheet

The equipment flowsheet used in the plant is shown in Fig. 4.2.

5.0 PROCESS PERFORMANCE

5.1 Flash Mixer

The performance of the flash mixer was adequate during the run. Optimum agitator speed was 400 rpm.

5.2 Flocculation

During start up lumps of sludge and algae floated to the surface of the solution in the flocculator carrying quantities of the newly formed iron floc. This condition was corrected by periodically raising the liquid level to the overflow point to float out the buoyant material. As was pointed out in Section 4.1, this condition was probably caused by the emptying of the "dead leg" on the suction side of the Lime Soda Plant feed pump. After 8 hours of operation the condition was corrected.

Rotation of the paddle agitator remained at 1.5 rpm throughout the run. The indicated pH of the solution in the 280-gallon flocculator vessel averaged 11.7; analysis of the solution by a Beckman pH meter gave values which averaged 11.8.

5.3 Clarifier

A total of 97 hours of continuous operation was completed using the 1870-gallon clarifier vessel. Of this total, 24 hours was required to displace the sludge formed during the previous run and to establish equilibrium. The average hardness (as CaCO₃) of the clarifier effluent during the run was 31.6 ppm accompanied by a turbidity of 2.6 ppm.

5.4 Polishing Filters

The sand filter remained in service for the entire run period, the pressure drop increasing 3.3 psi as solids accumulated on the bed. Hardness and turbidity of the filtrate were reduced by factors of 14 and 9, respectively, yielding solution containing 2 ppm of total hardness (as CaCO₃) and 0.29 ppm turbidity.

5.5 Ion Exchange

A total of 1564 bed volumes of treated waste water was passed through the 10-in diameter by 8-ft resin bed. Effluent discharged from the unit contained

Fig. 4.2. Preliminary flowsheet for low-level waste-treatment pilot plant.

- 7 -

UNCLASSIFIED ORNL-LR-DWG 59977 R1

no detectable hardness; the turbidity was 1.98 ppm which was due to lightly colored organic substances leached from the resin. The average pressure drop across the resin bed was 19.7 psi with neglible increase throughout the operating period.

The radiation level of the column increased to 1-2 mr/hr over the entire length of the bed.

Following the sorption step the resin was eluted with 10 volumes of $0.5~\underline{\text{M}}~\text{HNO}_3$ followed by a one volume rinse with demineralized water. The CS-100 carboxylic-phenolic resin was then converted to the Na form by passing 20 volumes of $0.1~\underline{\text{M}}$ NaOH through the bed. Elution curves for gross γ , Cs¹³⁷ and Sr⁹⁰ are presented in Fig. 5.5 and 5.6.

5.6 Sludge Filtration

Two methods of sludge removal were followed; in one, a fully-contained rotary drum Oliver filter was used that was barely capable of holding the sludge blanket level constant during equilibrium conditions, and in the other series of three 55-gallon drum decantation vessels were used in conjunction with the rotary filter during transient conditions.

Operability tests with non-radioactive feed indicated that the rotary filter will handle those solids produced by the addition of 5 ppm Fe. The use of 10 ppm Fe plus the additional solids present in the process waste water made the use of the decantation vessels necessary during part of run HR-2.

Feed to the rotary drum unit contained an average of 10.7 g of bone dry solids per liter of sludge; the feed rate was varied between wide limits in efforts to maintain the sludge blanket at the desired depth. The sludge cake discharging from the filter contained 87 per cent moisture; radiochemical analysis indicated 74 d/m of ${\rm Co}^{60}$ and 147 d/m ${\rm Sr}$ (< 5% ${\rm Sr}^{89}$) per mg of dry solids and lesser quantities of TRE.

6.0 REFERENCES

- 1. J. T. Roberts and R. R. Holcomb, "A Phenolic Resin Ion-Exchange Process for Decentaminating Low-Radioactivity-Level Process Water Wastes," presented at the American Chemical Society Meeting, New York, September 11-16, 1960, CF-60-8-103.
- 2. R. E. Blanco and E. G. Struxness, "Waste Treatment and Disposal Progress Report for April and May, 1961," CF-61-7-3, August 1, 1961.
- 3. J. M. Holmes and W. R. Whitson, "Design of a Low Level Pilot Plant for Scavenging-Ion Exchange Process," (in preparation).



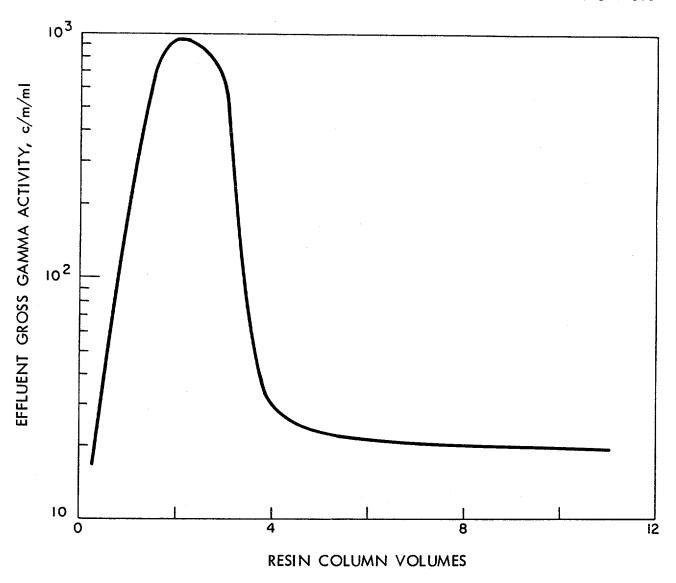


Fig. 5.5. Gross gamma elution of Duolite CS-100 resin with 0.5 M HNO3. Run HR-2.

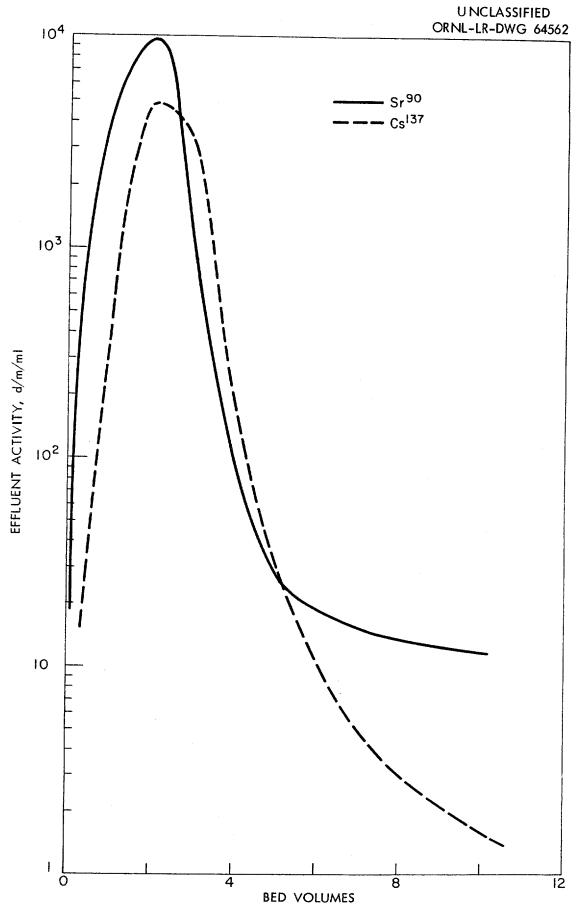


Fig. 5.6. Removal of Cs 137 and Sr 90 from Duolite CS-100 resin with 0.5 \underline{M} HNO3. Run HR-2.

7.0 APPENDIX
Table 7.1 Low Level Waste Treatment - HR-2

		Ion	Exchange	10.00	۰	•	۰	۰	•	11.80	11,75	11.70	11.80	11.60	08 [1	ָ ה ה	٠ ا ا	אליר א	20°11	11.85	11,80	11.85	11.85	11,85	11.90	11.82	11.85	11.85	11,85	11.85	11.90
Hu	***		Clarifier							11.90				11.60			•	•	1.80			•	•	•	•		•		۰	11.90	
		Floccu-	lator	11.90		11.85 RF	•	77.00	۰	11.80	11.80	11.75	•	11.60		•		•	11.85	, 8, 7, 6,	,8,	•	•		٠.				•	11.85	11.90
			Feed			· (-	-	-	-	-	7.35	. •																		7.45	
m)	Ion	Exchange	Column	Н	Q	. –	1 ~	- 1 (۰	p.5	۰	αı	ત્ય	-	ત	۱۴۰) (°	ب ئ		3. 5.	٦	7	0	႕	Н	ന	_	_	٦, ارن	_
Turbidity (ppm		Polishing	Filter	٦	-	0.5	•	-i -c	Э :	0	0	0	0	0	0	0	0	0	0	ч	н	•	0.5	0	0	0	0	0	0	0 0)
Tur			Clarifier	†	7.	4.5	,	ე –:	‡ (m	m		р. 5	ત્ય		1.5		a	4.5		2.5	ત	ଧ	~	Н		ู้ เรา	_	CU	લ લ	V
	Ion	Exchange	Column	0	0	0	C			→ · ✓ ·		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0 0	>
as CaCO ₂)		Polishing	Filter	a	,- -	Н			- - \	-																				o c	
Hardness (ppm s			Clariner	36	33	5¢	22	9	gα	Q (מא ל	ည္က	27	30	30	58	83	38	C 1 1	41	45	47	ဗ္တ 🤅	တ္တင့်	ჯ წ	, 100 100 100 100 100 100 100 100 100 10	<u></u>	34	34	35	7
Ha		<u>[</u>	reed	120	120	118	124	מטר	ן נכן	ט ל	בי קיני	011	OTT	120	118	120	118	120	118	118	118	1.14 2.10	25	02T	150	120 20 100		124	122	0 0 0 0 0 0	aller fores
	i	Time,	LIL	1.5	•	7.0	10.0	13.0	֝ ֓֓֞֜֜֜֝֓֓֓֓֓֓֓֓֓֓֡֓֡֓֓֓֡֓֡֓֓֡֓֡֡֡֓֡֓֡֓֡֓֡֓֡) () V	N - K		26.5	•	35.	35	38	47	5,	운.	₹ 2	Ţί	47	57	ر ا ا	ו ע	ひ へ の	0 5	7)	<u> </u>

DISTRIBUTION

- 1. R. E. Blanco
- 2. J. O. Blomeke
- 3. J. C. Bresee
- 4. R. E. Brooksbank
- 5. K. B. Brown
- 6. F. R. Bruce
- 7. K. E. Cowser
- 8. F. L. Culler
- 9. D. E. Ferguson
- 10. H. E. Goeller
- 11. R. R. Holcomb
- 12. J. M. Holmes
- 13. W. H. Jordan
- 14. E. Lamb
- 15. L. C. Lasher
- 16. W. H. Lewis
- 17. W. T. McDuffee
- 18. J. T. Roberts
- 19. E. G. Struxness
- 20. J. C. Suddath
- 21. M. E. Whatley
- 22. E. J. Witkowski
- 23. Laboratory Records ORNL RC
- 24-25. Laboratory Records